

# INCREASING THE VELOCITY OF ALKALI ACTIVATED REACTIONS USED IN SOIL STABILISATION

## AUMENTO DA VELOCIDADE DAS REAÇÕES DE ATIVAÇÃO ALCALINA USADAS NA ESTABILIZAÇÃO DE SOLOS

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### ABSTRACT

Alkali activated materials based on aluminosilicates, such as fly ash (FA), are regarded as important alternatives to Ordinary Portland cement (OPC), not only by reducing CO<sub>2</sub> emissions, but also by his enhanced mechanical behaviour. The alkaline activation of fly ash has been recently used in soil stabilization with promising results. However, the extension of the curing process may not be fast enough in situations demanding higher strength levels at early stages. Higher curing temperatures, common in ideal laboratory conditions, are not viable in field geotechnical applications. Therefore, alternative ways to increase the rate at which the alkaline activation reactions occur at ambient temperature need to be developed. The use of hydrated lime (HL) emerges as a possible solution. In that sense, different mixtures of soil, FA, HL and water or alkaline solution were prepared and tested in unconfined compression at 3, 7 and 28 days of curing. Alkali activated mixtures showed higher strength, which was further increased when HL was included.

### RESUMO

Materiais ativados por soluções alcalinas como as cinzas volantes (FA) são considerados importantes alternativas ao cimento Portland não apenas por reduzirem as emissões de dióxido de carbono como também pelo seu comportamento mecânico melhorado. A ativação alcalina de cinzas foi usada recentemente na estabilização de solos com resultados promissores. No entanto, o desenvolvimento do processo de cura pode não ser suficientemente rápido em situações que necessitem de elevada resistência em idades jovens. O aumento da temperatura surge como alternativa de contornar este problema, mas se tal é fácil de implementar em laboratório não é viável em aplicações geotécnicas de campo. Assim, são necessárias soluções alternativas para aumentar a velocidade a que as reações de ativação alcalina ocorrem à temperatura ambiente. O uso de cal hidratada (HL) surge como uma solução possível. Nesse sentido, diferentes misturas de solo, FA, HL e água ou solução alcalina foram preparadas e testadas em compressão simples aos 3, 7 e 28 dias de cura. As misturas com ativação alcalina apresentaram uma maior resistência, a qual foi ainda melhorada quando HL foi adicionada.

### 1 - INTRODUCTION

The concept of alkali activated materials (AAM) as an alternative to Portland cement has been known since at least 1908. However, fundamental research has blossomed internationally after 1990 (Provis and van Deventer, 2014). The interest on AAM for soil stabilisation is even more recent, but promising results have been reported (Zhang et al., 2013; Sukmak et al., 2013; Cristelo et al., 2011, 2012, 2013; Silva et al., 2013; Rao and Acharya, 2014; Rios et al., 2016; Phummiphan et al., 2016). In particular, the use of fly ash in AAM has attracted attention due to the possibility of including a waste by-product without generating carbon dioxide emissions as in Portland cement production (Cristelo et al., 2015).

The reaction of a solid aluminosilicate with a highly concentrated aqueous solution produces a synthetic alkali aluminosilicate material with binder properties. This binder is dominated by an alkali aluminosilicate gel whose structure is known to be closely related to the precursor gels observed as intermediates during hydrothermal synthesis of zeolites from the same aluminosilicate solids. Crystalline zeolites and related materials are developed over time, with higher temperatures and higher water contents favouring higher crystallinity (Provis et al., 2014). In fact, in the alkaline activation reaction the minimum quantity of alkaline activator is used to provide enough workability to the mixture, so the water content is generally much lower than in zeolite formation.

To maximize the reactions that generate the AAM, aluminosilicates that have suffered a previous thermal treatment, involving the loss of water and changes in the coordination of aluminium and oxygen ions, are preferred. As a consequence of this treatment the aluminosilicate loses its crystalline structure, becoming more entropic and more available to react. Aluminosilicates such as slags, fly ashes, volcanic ashes or dust from bricks or tiles are examples of good materials to be alkali activated due to their known thermal history. The cooling stage, which is generally fast, reinforces their amorphous structure and thus their reactivity. This makes their structure rather closed with low permeability. If the curing process is performed above the room temperature, the structure becomes even more impervious at atmospheric pressure conditions. For that reason, thermal or steam curing is usually applied to alkali activated binders as strength development is slow at room temperature (Andini et al., 2008).

The activator solutions generally used are sodium or potassium hydroxide, sodium carbonate, sodium or potassium silicate, and very often mixtures of these compounds. The type, amount and concentration of the activator has to be studied for each aluminosilicate, since the chemical composition, and fineness greatly affect the activation reaction. Natural materials do not have the "optimum" chemical composition, so, it is usually required to mix different components. Some materials are rich in silica, other in alumina, being therefore necessary to perform a composition study to optimize the results. The activators can be divided in two groups: simple and complex. The simple ones are made from an alkaline base such as sodium or potassium hydroxide. The complex ones are formed by the association of an alkaline base with a sodium or potassium silicate.

AAM are very sensible to curing conditions. Although these conditions are not very difficult to obtain, it is very important to respect them. Temperature and humidity should be well controlled and it is also necessary to avoid the possibility of shrinkage associated to the loss of water. Curing and strength can be highly accelerated with temperature, and significant strength values have been reported by several authors when curing at 70°C or 85°C (e.g., Palomo et al., 1999; van Jaarsveld et al., 2002). On the other hand, shrinkage can be reduced if solid particles are added to the mixture because the solid structure is able to absorb part of the stresses due to the volumetric contraction.

In the few literature works reporting mixtures of AAM with soil the following can be found: low calcium fly ash AAM curing at high temperatures between 65° and 85°C (Sukmak et al. 2013), or high calcium fly ash AAM curing at lower temperatures around 30°C (Phummiphan et al., 2016), or metakaolin AAM curing at even lower temperatures around 23°C. These examples emphasize the idea that temperature has a very significant effect. However for field geotechnical applications high curing temperatures are not feasible. On the other hand, there are situations requiring high strength at early ages, for example due to constraints related to road serviceability which demand short construction periods. It is therefore important to search for alternative ways of increasing the curing rate.

Some authors (Ghosh and Subbarao, 2001; Kumar et al., 2007; Samaras et al., 2008; Cristelo et al., 2009; Consoli et al., 2011) indicated that significant improvement in soil strength (especially clayey soils) can be found adding lime and fly ash as it enhances the pozzolanic reaction of the mixture. Therefore, it may be possible to obtain the same effect with activated ash increasing the velocity of alkaline activation reactions.

In this paper, different mixtures prepared with soil, lime and fly ash (activated or non activated) were compared in terms of the evolution of strength and stiffness with curing time. This enabled to understand the advantage or disadvantage of using those binders alone or combined.

## **2 - MATERIAL AND METHODOLOGY**

### **2.1 - Test materials**

The soil involved in this experimental program is a silty sand from Poland classified as SC-SM according to ASTM (2011) D 2487-11. The fine fraction of the soil is 43.8% and its plasticity index is 6.5% given by a liquidity limit of 19.5% and a plasticity limit of 13%. Considering a uniformity coefficient of 167 and a curvature coefficient of 6.7 the soil is assumed to be well graded. Compaction properties given by Modified Proctor tests indicate an optimum water content of 8.2% and a maximum dry density of 21.29 kN/m<sup>3</sup>.

Low calcium fly ash – FA (classified as class F according to ASTM (2015) C 618) was used which was provided by a Portuguese thermo-electric power plant.

Hydrated lime (HL) provided by the company Lusical ([www.lhoist.com/pt\\_en](http://www.lhoist.com/pt_en)) was also used.

The activator solution used was composed by sodium hydroxide (SH) and sodium silicate (SS). The former was supplied in pellets, with a specific gravity of 2.13 at 20°C (99 wt.%), which was then dissolved in water to predetermined molal concentration of 12.5. The sodium silicate had a unit weight of 1.464 g/cm<sup>3</sup> at 20°C, a SiO<sub>2</sub>/Na<sub>2</sub>O weight ratio of 2.0 (molar oxide ratio of 2.063) and a Na<sub>2</sub>O concentration in the solution of 13.0%. Deionised water was used in every mixture prepared during the work presented. A sodium silicate to sodium hydroxide ratio of 0.5 was used for all the mixtures where an activator solution was added.

## 2.2 - Specimen preparation

The soil was first dried and de-flocculated by hand before the preparation of the specimens. The solids (soil, fly ash and/or lime) were then dry mixed in a Hobart counter mixer, and the liquid phase was carefully added requiring an additional 10 min mixing period. The resulting mixture was transferred to a stainless steel mould and compacted in specimens of 70 mm of diameter and 140 mm height. The moulds were stored at a temperature of 20°C ± 1°C and a relative humidity of 90% ± 3% wrapped in cling film to avoid moisture loss. Forty-eight hours after compaction the specimens were demoulded and left to cure in the same conditions.

## 2.3 - Experimental plan

Table 1 presents the studied mixtures, indicating the mass percentage of each component of the solid phase, as well as activator/fly ash ratio and the water content of the mixture for the characterisation of the liquid phase. The mixtures were identified using the following code: 'S' (soil); 'A' (fly ash); 'L' (lime) and 'AA' (alkali activated). Four different mixtures with soil/precursor weight ratios of 95/5, 90/10, 80/20 and 75/25 were prepared using deionized water; and additional three mixtures, with weight ratios of 85/15, 80/20 and 75/25, were prepared using alkaline activator at an activator/ash ratio of 0.7. Different precursor compositions were considered by combining FA and HL in different proportions, and every specimen was moulded with a dry unit weight of 18.0 kN/m<sup>3</sup>. For the seven mixtures identified in Table 1 tests were made at three different curing periods: 3, 7 and 28 days. Since three equal specimens were moulded for each mixture in order to have representative results, a total of 21 specimens of stabilised soil were prepared plus three soil specimens.

Table 1- Identification of the moulded mixtures

Mixture	Solid Phase			Liquid phase	
	Soil (%)	Fly Ash (%)	Lime (%)	Activator/Fly ash (wt.)	Water content (%)
Soil	100				5
S.L1	95	-	5	-	5
S.L2	90	-	10	-	5
S.AA	85	15	-	0.707	8
S.AL1	80	15	5	-	5
S.AL2	75	15	10	-	5
S.AAL1	80	15	5	0.707	8
S.AAL2	75	15	10	0.707	8

The unconfined compressive strength (UCS) test was used to assess the performance of the mixtures, by means of an Instron® electro-mechanical testing rig, fitted with a 50 kN load cell, as illustrated in Figure 1. The tests were carried out under monotonic displacement control, at a rate of 2 mm/min, and the entire stress-strain curve was obtained from each test.



Figure 1 – Unconfined compression strength testing apparatus

### 3 - TESTS RESULTS

The results were analysed in terms of the unconfined compression strength and the secant modulus at 50% of the maximum deviatoric stress ( $E_{50}$ ). This last parameter is essential in some constitutive models included in finite element codes, and therefore its calculation is extremely useful for design purposes. Figure 2 shows some stress-strain curves and a determination of  $E_{50}$ , as an example of what was performed for the other mixtures. In particular, Figure 2a represents the stress-strain curves of the soil and the mixtures with 5% of lime content at 28 curing days, where it is clear that the highest strength is provided by the activated ash with lime. It is also noticed that adding lime to the ash slightly improves the strength. Figure 2b shows the evaluation of  $E_{50}$  for the mixture of soil and 5% of fly ash (S.AL1).

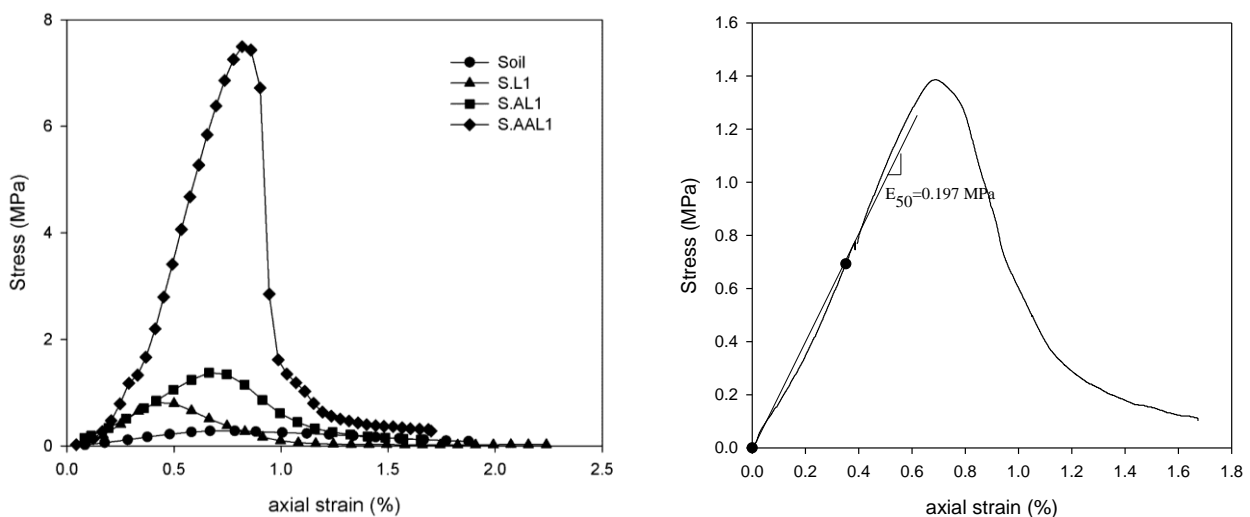


Figure 2 – Stress-strain curves for the mixtures with 5% of lime at 28 curing days (a), and stress-strain curve for the mixture S.AL1 indicating the evaluation of  $E_{50}$  (b)

In Figures 3 and 4 the evolution of strength and  $E_{50}$  with curing time is presented for all the mixtures. With a few exceptions, the order of magnitude of the stiffness and strength values agrees well, i.e. the mixtures with the lower and higher strength values presented also the lower and higher stiffness values, respectively. Also significant is the strength increment between the 7<sup>th</sup> and 28<sup>th</sup> day mark, during which the mixtures

that included lime and activated ash showed better performance than the others. The presence of fly ash in the mixtures prepared with water promoted only a slight beneficial effect in strength. On the other hand, the addition of lime showed a significant influence on the UCS for both activated and non activated fly ash mixtures, but especially in the latter. As expected, mixtures containing both fly ash and alkaline activator had a steady increase of mechanical strength over time, which was further increased when lime was also included. Based on the results it was concluded that the presence of fly ash and, in particular, lime has a significant influence on the mechanical response of the mixtures.

The presence of lime is important to increase the strength gain rate of the mixtures, therefore fulfilling the aim of this research. Lime can accelerate the chemical reactions of alkaline activation leading to a higher strength almost from the beginning of the reaction. This means that it may be possible to use AAM for soil stabilisation in field geotechnical applications at ambient temperature.

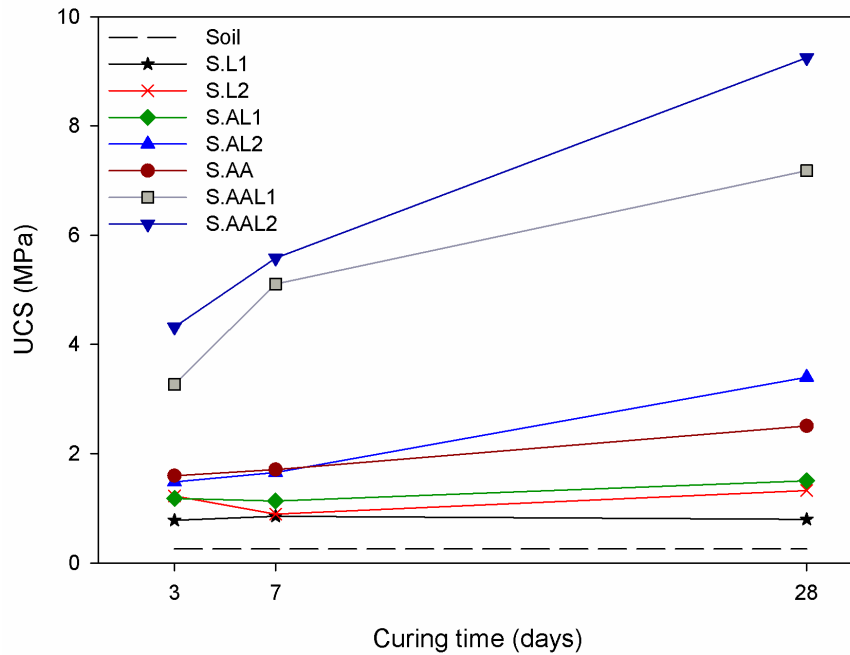


Figure 3 – Evolution of the unconfined compression strength with curing time

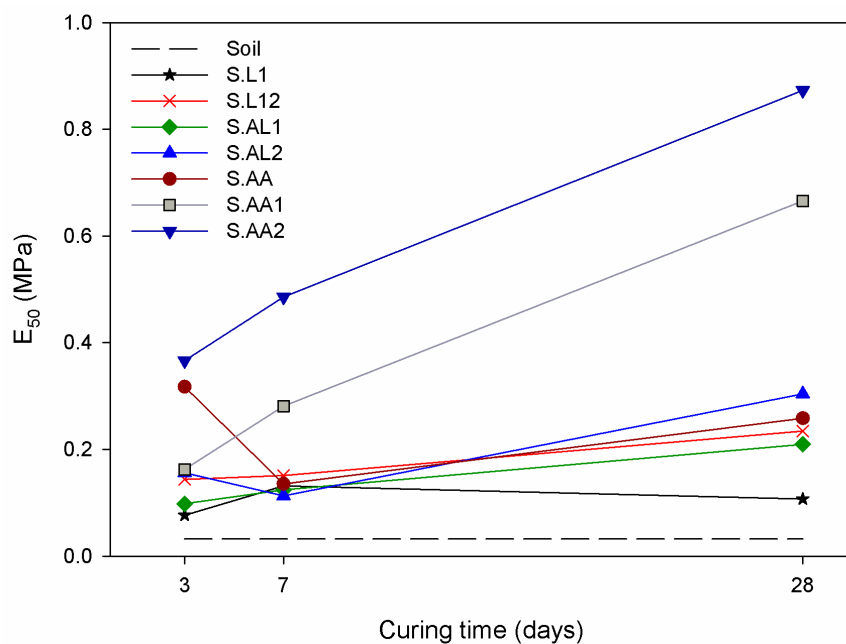


Figure 4 – Evolution of the secant Young modulus at 50% of the maximum deviatoric stress ( $E_{50}$ ) with curing time

All the specimens exhibited a failure mode typical of cemented materials (a sub-vertical shear plane) as it is illustrated in Figure 5, indicating that even the mixtures with low strength have strong bonds between particles.



Figure 5 – Photograph of the tested specimens

#### 4 - CONCLUSIONS

Alkali activated materials (AAM) have been studied for some years showing that they can be a viable alternative to Portland cement for different applications. In particular the use of fly ash based AAM to produce concrete has attracted attention due to its great advantage of using a waste by-product from thermo-electric power plants that if not used is landfilled occupying enormous areas and creating significant environmental problems. Another advantage it to avoid the emissions of high amounts of carbon dioxide to the atmosphere associated to the production of Portland cement. Finally, the concrete from AAM has shown good mechanical and durability properties, which lead to some studies on the use of AAM for soil stabilization. Although promising results have been reported in the literature most of them involve the use of high curing times, high curing temperatures, or expensive aluminosilicates such as metakaolin which are not feasible in field geotechnical applications.

In this paper, results were presented to evaluate the possibility of using hydrated lime to improve and accelerate the chemical reactions associated to the alkaline activation of fly ash at room temperature. For that purpose, different mixtures of soil, fly ash, lime and water or alkaline solution were prepared to be tested in unconfined compression tests after different curing periods namely 3, 7 and 28 days. This enabled to understand the advantage of adding lime to the soil-ash mixtures in activated or non activated conditions.

The results indicated that lime slightly increases the strength of soil and non activated ash mixtures, but increases significantly the strength of alkali activated soil-ash mixtures. In fact, these mixtures presented the best results achieving (for 10% lime content) 16 times the soil strength at 3 days, and 35 times the soil strength at 28 days.

From these results, it can be concluded that lime can be used satisfactory with AAM enhancing the soil behavior at room temperature since it accelerates the chemical reactions of alkaline activation leading to a higher strength almost from the beginning of the reaction. The fact that lime proved to be very effective in increasing the strength gain rate in alkali-activated fly ash shows a promising solution to overcome what is one of major concerns regarding the use of alkali activation for soil improvements.

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